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④ INTERFEROMETRIC METHODS FOR DEVICE FABRICATION.

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Description**Background of the invention****1. Field of the invention**

The invention relates to processes of device fabrication including interferometric techniques for measuring or monitoring the thickness of layers.

2. Art background

The fabrication of devices such as information processing devices, e.g., integrated circuit devices, magnetic bubble devices, and integrated optics devices, involves etching patterns into regions, e.g., layers, of substrate material of different composition which are either incorporated into the device undergoing fabrication or removed during the fabrication process. Regions which are removed include, for example, layers of organic polymer resist. Such a resist layer is typically patterned by being exposed to actinic radiation through a mask, and then subjected to either a wet or dry etchant which selectively etches either the exposed or the unexposed portions. Regions which are incorporated into devices include, for example, layers of semiconductor material, metal, and dielectrics, e.g., silicon dioxide. Typically, a pattern is etched or milled into one of these layers by initially forming an etch or milling mask such as a patterned resist, on the layer, and then etching or milling the uncovered portions of the layer with a wet or dry etchant or a milling agent. Hereafter, for the sake of brevity, it is to be understood that the term "etching", as used in relation to materials other than resists, encompasses milling.

An important consideration in these etching procedures is control of etch depth. For example, overetching a layer of resist material (subjecting the layer to an etchant for a longer period of time than is necessary to etch through its thickness) is generally undesirable since this often leads to a loss of linewidth control during pattern replication, e.g., when using the resist as an etch mask. On the other hand, it is, at times, desirable to terminate etching at a desired depth within a homogeneous layer of material, or at the interface between two different layers of material, to be incorporated into a device.

Various techniques have been devised for monitoring etch depth. One of the most widely used (now conventional) etch depth monitoring techniques relies on the transparency to visible light (light having a wavelength ranging from about 400 nm to about 700 nm) of many substrate layers. (Such a layer is transparent to incident visible light if it transmits at least 5 percent of the incident visible light). In accordance with this technique, visible light from, for example, a laser, e.g., a helium-neon laser (which emits light having a wavelength of 632.8 nm), is directed onto a bare, i.e., uncovered, area of the transparent (to the visible light) layer undergoing etching, and the intensity of the light reflected from the layer is detected and recorded as a

function of time. Because the layer is transparent, the incident light is both reflected from the upper surface of the transparent layer and is transmitted, i.e., refracted, through the layer, as shown in Fig. 1. If the layer overlies a reflective surface, then the refracted light is also reflected upwardly through the layer, exiting the layer to interfere with the light reflected from the upper surface of the layer. As etching proceeds, the thickness of, and thus the optical path length through, the substrate layer being etched is reduced. Consequently, at specific thicknesses, destructive or constructive interference, which correspond to, respectively, a relative minimum and relative maximum in the recorded intensity-time curve, occurs. It is possible to relate to time intervals between these intensity extrema to changes in etch depth.

A primary reason for the wide use of the above-described technique is its compatibility with the conventional alignment procedure. That is, fiducial marks in substrates are employed to align resist exposure masks. The resist layer is formed over the substrate and thus commonly over the fiducial mark. The exposure mask is aligned with the fiducial mark by shining visible light (generally the very same wavelength of visible light used for etch monitoring) onto the resist layer (which, because of its transparency to the incident visible light, permits the fiducial mark to be detected). Since the ability to detect fiducial marks with visible light is considered essential, changes in the etch monitoring technique involving increases in the opacity of substrate regions overlying these marks are avoided.

While the conventional etch monitoring technique has many advantages, including compatibility with the conventional alignment technique, difficulties arise when the substrate contains more than one transparent region. For example, if a transparent substrate region undergoing etching is supported by a second transparent region which overlies a nonplanar surface, e.g., a stepped surface, then incident visible light will be refracted through both transparent substrate regions, reflected from the structures in the non-planar surface, and transmitted upwardly through the two transparent regions. Interactions between light beams which have so been refracted and reflected often produce an unwanted interference signal which, in many cases, is so large that the interference signal from the substrate region undergoing etching is undetectable.

The conventional etch monitoring technique has other difficulties. For example, if the etch rate (of the region being etched) is constant, then the etch end point, i.e., the instant in time when the interface between the transparent substrate region undergoing etching and the underlying region is reached, corresponds to a frequency change of the intensity oscillations in the recorded intensity-time curve. But because this frequency change can, and does, occur at any point along an intensity oscillation (in the intensity-time curve), the etch end point is difficult to

anticipate. Moreover, if the thickness of the substrate layer undergoing etching is less than, or is a relatively small multiple of, the etch depth change corresponding to the spacing between adjacent intensity extrema, then there will be less than one-intensity oscillation, or a relatively small number of intensity oscillations, corresponding to the substrate region thickness. These two effects often make it difficult to accurately determine etch end point, which often results in undesirable underetching or overetching of the region being etched.

In an alternative etch monitoring technique, applicable to opaque or transparent (to visible light) substrate regions, visible light is directed onto an area of a substrate region (undergoing etching) shielded by a patterned etch mask. The incident visible light is reflected both from the etch mask surface and from the etch pit (or pits) being etched into the substrate region. At specific etch depths, there is either constructive or destructive interference, with consequences similar to those described for the previous procedure.

The alternative etch monitoring technique also has many advantages and is also compatible with the conventional alignment technique (the etch mask is typically transparent to incident visible light). However, if the (transparent) etch mask itself undergoes etching during the etching process (as is often the case), then this results in an interference signal (produced by varying interactions between light beams reflected from the top and bottom of the etch mask) unrelated to the etching of the substrate region. This unrelated signal is often much larger than that associated with the etching of the substrate region, which again results in undesirable underetching or overetching.

The etch end point of a substrate region (the point in time when the region has been etched through its thickness) is readily determined if the thickness, and etch rate, of the substrate region are known (for example, if etch rate is constant, then the etch time required to achieve etch end point=thickness/etch rate). Thus, techniques have been devised for measuring the thickness of substrate regions. One such thickness measurement technique, which is widely used because it, too, is compatible with the conventional alignment technique, involves shining visible light onto the (transparent) substrate region whose thickness is to be measured. If the region is known to have a thickness less than $\lambda/4n$, where λ is the wavelength of the incident light, and n is the index of refraction of the layer, when the thickness is readily determined from the intensity of the reflected light. (See, for example, O. S. Heavens, *Optical Properties of Thin Films* (Dover Publications, New York, 1965), Section 4.4). Alternatively, the thickness is determined by shining visible light of different wavelengths onto the region (whose thickness is being measured), and measuring the intensity of the reflected light for each wavelength. At specific wavelengths, interference phenomena occur through the pre-

viously described mechanisms. The thickness of the substrate region is readily calculated from the observed intensity extrema as shown in, for example, F. Reizman and W. van Gelder, *Solid State Electronics*, Vol. 10, p. 625 (1967).

While the above thickness measurement technique has been found to be useful in many instances, difficulties arise when measuring the thickness of a relatively thin, transparent (to the incident visible light) region formed on, e.g., deposited onto, a relatively thick, transparent region. Typically, the thickness of the relatively thick region is first measured. Then, the relatively thin region is formed on the thick region and the combined thickness of the two transparent regions is measured. Finally, the thickness of the relatively thick region is subtracted from the combined thickness to determine the thickness of the relatively thin region. However, if the measured thickness of the relatively thick transparent region is in error even by a relatively small amount, a substantial error in the measured thickness of the relatively thin region often occurs. As a consequence, the relatively thin region often suffers undesirable underetching or overetching.

Thus, more accurate etch monitoring and thickness measurement techniques continue to be sought.

IBM Technical Disclosure Bulletin, vol. 24, No. 9, Feb 1982, pages 4804—4805 discloses an etch rate monitor comprising a thin film of boron-doped silicon stretched on a silicon frame. A layer of the same material as the layer to be etched is formed on the thin film (unless the material to be etched happens to be the same as that of the thin film). A further layer may be applied beneath the thin film to increase the reflectivity at its bottom surface.

In the methods to which the present invention relates, the measuring or monitoring is one of the processing steps carried out on a body to fabricate a device.

45 Summary of the invention

In a method as set out in the claims, the light used for the monitoring or measuring is light to which the second region is substantially opaque. This light may be outside the visible range, and, in particular, may be ultra-violet, depending on the materials used. The presence of the opaque region precludes transmission through the opaque region of reflections of refracted incident light from underlying surfaces or regions, and thus precludes the formation of unwanted interference signals. Consequently, etch depth, or thickness, is more accurately determined.

A variety of substrate regions are substantially opaque to nonvisible electromagnetic radiation, e.g., ultraviolet light. Thus, in one embodiment of the invention, a substrate region which is, for example, to be incorporated into a device, is made substantially opaque to the incident light by using nonvisible incident light. In other embodiments of the invention involving a substrate

region which is, for example, a sacrificial coating, the substrate region is made substantially opaque to the incident light by using, for example, nonvisible incident light. Alternatively, the desired opacity of the sacrificial substrate region is achieved by incorporating a light-absorbing material which does not absorb the light used for alignment purposes, into the substrate region and using an incident light the nonvisible or visible light (whose wavelength or wavelength range is different from that used for alignment purposes) absorbed by the light-absorbing material. Thus, improved accuracy is achieved while maintaining compatibility with the alignment procedure.

Brief description of the drawing

The invention is described with reference to the accompanying drawings, wherein:

Fig. 1 depicts a technique for monitoring etching;

Fig. 2 depicts, in cross section, the tri-level resist;

Fig. 3 schematically depicts an apparatus for monitoring etching and measuring thickness;

Figs. 4-6 depict intensity-time curves obtained during the etching of a substrate using both the inventive and conventional etch monitoring techniques; and

Fig. 7 depicts an intensity-wavelength curve obtained using both the inventive and conventional thickness measurement techniques.

Detailed description

The etch monitoring and thickness measurement techniques employed in the inventive device fabrication method are generally similar to the previously used techniques in that they involve shining light onto a portion, or all, of a substrate region of interest, and detecting the intensity of all or a portion of the reflected light. However, the former techniques are distinguished from the latter techniques in that the incident light is chosen so that a substrate region (or regions) underlying, and/or a patterned substrate region (or regions) overlying, the substrate region of interest is substantially opaque to at least a portion of the incident light. (For purposes of the invention, an overlying patterned substrate region is one which overlies only a portion or portions, but less than all, of the substrate region of interest. Such a patterned region is formed, for example, by removing selected portions of a substrate region which overlies all of the substrate region of interest, or by a selective deposition of overlying substrate region material onto the substrate region of interest or an intervening substrate region). Generally, the incident light is also chosen so that the substrate region of interest is substantially transparent to at least a portion of the incident light. For purposes of the invention, a substrate region is a material region of a substrate which is either incorporated into the device undergoing fabrication, or removed during the device fabrication process. In addition, a substrate region is substantially opaque to any

portion of the light incident on the region provided less than about 5 percent of that incident portion is transmitted through the region. On the other hand, a substrate region is substantially transparent to any portion of the light incident on the region provided more than about 5 percent of that incident portion is transmitted through the region.

The purpose of a substantially opaque substrate region underlying a substrate region of interest, e.g., a substrate region whose etching is being monitored or whose thickness is being measured, is to prevent the transmission (through the opaque region) of light reflected from surfaces and structures underlying the opaque region. On the other hand, the purpose of a substantially opaque, patterned substrate region overlying a substrate region of interest, e.g., a substantially opaque, patterned resist layer overlying a substrate layer whose etching is being monitored, is to prevent the transmission (through the opaque region) of light reflected from the interface between the opaque region and an underlying region, e.g. the region of interest. Consequently, undesirable signals not associated with the etching or thickness of the substrate region of interest are largely, or entirely, eliminated. Thus, the accuracy in the determination of etch depth, or the thickness, of the substrate region of interest is significantly improved.

The presence of a substantially opaque substrate region underlying a substrate region of interest is advantageous for reasons other than the avoidance of undesirable interference signals. For example, when monitoring the etching of a substantially transparent substrate region, the presence of a substantially opaque substrate region immediately beneath the substrate region undergoing etching has the unexpected advantage that the etch end point (which corresponds to a change in the frequency of the intensity oscillations in the intensity-time curve) always occurs at a relative maximum or a relative minimum in the intensity-time curve. Thus, etch end point is more readily anticipated, which permits greater accuracy in the determination of etch end point than was previously possible.

The choice of the incident light is determined by the wavelength (or wavelengths) of light absorbed by the substrate region overlying or underlying the substrate region of interest and, if so desired, transmitted by the substrate region of interest. For example, if the overlying or underlying region inherently absorbs more than about 5 percent of the light (to achieve substantial opacity) of a particular wavelength (or wavelengths), then the inherently absorbed light is used as the incident light (provided the substrate region of interest is substantially transparent to this incident light, if so desired). Generally, a useful wavelength of light (one inherently absorbed by the underlying or overlying substrate region and, if so desired, transmitted by the substrate region of interest) is found by forming, e.g., depositing, the substrate region of interest and the underly-

ing or overlying substrate region on separate substrates of known transmissivity to a particular wavelength of light. The two regions are then impinged with light of this particular wavelength, and the amount of light transmitted by the substrates is measured. In this regard, a wide variety of substrate materials are either substantially transparent or substantially opaque to nonvisible light, e.g., ultraviolet (UV) light (light having a wavelength ranging from about 150 nm to about 400 nm). For example, silicon dioxide is substantially transparent to UV light, while organic polymer resists (such as the resist sold under the trade name HPR-204 by the Hunt Chemical Company of Palisades Park, New Jersey) which have been subjected to a heat treatment at a temperature greater than about 210 degrees C, are substantially opaque to UV light.

The use of UV light, rather than visible light, for monitoring the etching of a substrate region also has direct consequences (other than the avoidance of undesirable interference signals) for the determination of etch end point. That is, when monitoring the etch depth of a substrate region undergoing etching, the spacing between two adjacent minima or two adjacent maxima in the recorded intensity-time curve corresponds to a change in etch depth proportional to the wavelength of the incident light. By using UV incident light, rather than visible incident light, the number of extrema in the intensity-time curve corresponding to a given etch depth change is increased (because the wavelength of UV light is less than that of visible light). Consequently, etch depth is more accurately monitored.

If the overlying or underlying substrate region does not inherently absorb more than about 5 percent of the light of any wavelength, or if it is desired to use incident light other than the inherently absorbed light, then a light-absorbing material (which does not absorb the wavelength used for alignment) is added to the region, provided this has no significant adverse impact on device operation. The incident light is then chosen to be the light absorbed by the light-absorbing material (provided the substrate region of interest is substantially transparent to this light, if this is desired). A light-absorbing material, is for example, a dye, e.g., a dye sold by the Pylan Packer Company of Garden City, New York under the trade name Morton Automate Blue 8, which absorbs light of wavelength equal to 632.8 nm.

It is convenient to differentiate those substantially opaque substrate regions which are ultimately incorporated into a device from those substantially opaque substrate regions which serve as sacrificial coatings removed during device fabrication. In the case of an underlying substrate region which is to be substantially opaque and which is to be incorporated into a device, the desired opacity is generally not achieved through the incorporation of light-absorbing material because this is usually difficult and often has adverse consequences for device operation. Rather, opacity is generally achieved

by choosing light of a particular wavelength or wavelength range to which the underlying substrate region is (inherently) substantially opaque and, if desired, the substrate region of interest is substantially transparent. Generally, the useful wavelengths are from the nonvisible, e.g., UV, wavelength range.

The incorporation of light-absorbing material into an underlying substrate region which serves as a sacrificial coating removed during device fabrication, generally has no adverse consequences for device operation. Thus, the desired opacity of such a region is achieved either by using the above procedure or by incorporating light-absorbing material into the underlying substrate region and choosing incident light which is absorbed by this material. For example, if the underlying substrate region is a sacrificial coating such as an organic polymer resist formed by conventional spin-deposition techniques, then a light-absorbing material is readily incorporated into the organic resist by dissolving the material in the resist solution (commercial organic resists are typically supplied in solution form). During spinning, the resist solvent evaporates leaving an organic resist material containing light-absorbing material. The incident light is then chosen to be the visible or nonvisible light which is absorbed by the light-absorbing material.

Generally, when the underlying substrate region is a sacrificial coating, the substrate region of interest, i.e., the overlying substrate region, is also a sacrificial coating because removal of the underlying region usually requires removal of the overlying region. Typically, the underlying and overlying substrate regions are material regions which serve as metallization or etch masks or regions of resist material of different composition.

In the case of an overlying, patterned substrate region which is incorporated into a device, light-absorbing material is generally avoided for the reasons given above. The desired opacity is usually achieved by using nonvisible, e.g., UV, incident light.

For an overlying, patterned substrate region which serves as a sacrificial coating, the incorporation of light-absorbing material generally has no adverse consequences for device operation. Thus, opacity is achieved by using incident visible or nonvisible light absorbed by the material. Alternatively, there is no incorporation of light-absorbing material, and the incident light is usually nonvisible, e.g., UV, light, for the reason given above. A sacrificial, overlying, patterned substrate region is, for example, a patterned etch mask which is substantially free of metallic atoms in their zero oxidation state, e.g., a patterned, organic polymer resist. For purposes of the invention, a material is substantially free of metallic atoms in their zero oxidation state provided it contains less than about 25 percent atomic concentration of metallic atoms in their zero oxidation state.

As a pedagogic aid to a more complete under-

standing of the invention, the application of the inventive etch monitoring technique to the etching of the silicon dioxide layer of the tri-level resist (regarding the tri-level resist see, for example, J. M. Moran and D. Maydan, *Bell System Technical Journal*, Vol. 58, pp. 1027—1038, (1979)), is described below.

The tri-level resist is generally (but not exclusively) used to etch very fine-line features (features having dimensions typically smaller than about 2 μm) into substrate layers having nonplanar, e.g., stepped, surfaces. As shown in Fig. 2, the tri-level resist includes a relatively thick layer 20 which covers, and planarizes, the nonplanar surface 10 of the substrate layer to be patterned. (The layer 20 planarizes the nonplanar surface 10 in the sense that the layer 20 covers the steps in the surface 10 and presents an essentially flat upper surface). Typically, the layer 20 consists of an organic polymer such as HPR-204 resist which is deposited by conventional spin-deposition techniques onto the nonplanar surface 10 (and subsequently baked). The thickness of the planarizing layer 20 ranges from about 0.5 μm to about 3 μm . A thickness less than about 0.5 μm is undesirable because such thin layers often have an undesirably large number of pinholes. A thickness greater than about 3 μm is undesirable because such thick layers require an undesirably long etch time, and often lead to a loss of linewidth control.

The tri-level resist typically includes a silicon dioxide layer 30 which overlies the planarizing layer 20. The layer 30 is deposited, for example, by conventional plasma deposition techniques. The thickness of the layer 30 ranges from about 0.08 μm to about 0.4 μm . Thicknesses less than about 0.08 μm or greater than about 0.4 μm are undesirable for the reasons given above.

A relatively thin layer 40 of resist, e.g., photoresist or e-beam resist or X-ray resist (depending upon the nature of the exposing energy), in turn covers the silicon dioxide layer 30. The layer 40 consists of, for example, the photoresist sold under the trade name AZ-2415 by the American Hoechst Corporation of Somerville, New Jersey, or the e-beam resist sold under the trade name PBS by Mead Technologies, Incorporated of Rolla, Missouri, or DCOPA X-ray resist sold by the Great Lakes Chemical Company of West Lafayette, Indiana. Generally, the layer 40 is deposited onto the layer 30 by conventional spin-deposition techniques and has a thickness ranging from about 0.3 μm to about 1.5 μm . A thickness less than about 0.3 μm is undesirable because such thin layers often have an undesirably large number of pinholes, while a thickness greater than about 1.5 μm is undesirable because it is difficult to resolve relatively small features (smaller than about 2 μm) with such thick layers.

In the course of etching a pattern into the nonplanar substrate layer, the pattern is first defined in the layer 40 by selectively exposing the layer 40 to actinic radiation (e.g., electromagnetic radiation or electron beams or X-rays) and

developing the layer 40. Then, using the patterned layer 40 as an etch mask, the pattern is transferred into the layer 30 by etching the layer 30 in, for example, a CHF_3 plasma. Thereafter, the pattern defined in the layer 30 is transferred into the layer 20 by etching the layer 20 in, for example, an O_2 plasma while using the layer 30 as an etch mask. Finally, the desired pattern is transferred into the nonplanar substrate layer by etching this layer while using the patterned layer 20 as an etch mask.

An important consideration in the above etching procedure is the need to avoid overetching of the silicon dioxide layer 30. Such overetching often results in degradation of the patterned resist layer 40, which in turn results in a loss of linewidth control and an increased possibility of defects, e.g., pinholes, during the etching of the layer 30 and, ultimately, similar consequences during etching of the nonplanar substrate layer. Attempts to monitor the etching of the layer 30 using conventional techniques (shining visible light on a bare portion of layer 30 or a portion covered by the patterned resist layer 40) have been thwarted by the transparency to visible light of layer 20. That is, the interference signal associated with the etching of the layer 30 is generally overwhelmed by a signal produced by refracted incident light reflected from structures in the nonplanar surface 10 and transmitted (upwardly, as viewed in Fig. 2) through the layer 20. But the etch monitoring of the layer 30 is readily and accurately achieved, in accordance with the invention, by, for example, using UV incident light to which the silicon dioxide layer 30 is substantially transparent and to which the HPR-204 layer 20 is substantially opaque. Alternatively, the Morton Automate Blue 8 dye, which absorbs visible light of wavelength equal to 632.8 nm, is incorporated into the layer 20 (as described above), and 632.8 nm wavelength light is used as the incident light during etch monitoring. The dye concentration (by volume) in the resist solution ranges from about 4 percent to about 10 percent. Concentrations less than about 4 percent are undesirable because they lead to opaque layers whose opacity is undesirably low. Concentrations greater than about 10 percent are undesirable because they often lead to opaque layers which exhibit undesirably low adhesion to the silicon dioxide layer 30 and the underlying substrate, and are relatively poor etch masks, i.e., are readily degraded during etching of the underlying substrate.

Subsequent to the etching of the silicon dioxide layer 30, the etching of the HPR-204 layer 20 is then readily monitored using, for example, incident visible light, because the layer 20 is substantially transparent to such incident light (provided any light-absorbing material incorporated into the layer 20 does not absorb the visible light). Moreover, the underlying substrate, if it is, for example, of silicon, will be substantially opaque to the incident visible light.

One aspect of the invention, i.e., the inventive

etch monitoring technique, is applicable, in general, to all etching techniques including, but not limited to, plasma and wet chemical etching, reactive sputter etching (also called reactive ion etching) and ion milling. In addition, the inventive method is also applicable to those techniques where a directed beam of energy or a directed beam of charged particles is used to directly pattern a substrate region, without the use of an etch mask.

The invention encompasses the application of the inventive etch monitoring and thickness measurement techniques to the fabrication of devices. That is a device such as an electronic information processing device is fabricated by a series of steps, well known in the art, which, for example, includes the step of etching a pattern just through the thickness of a substrate region. Alternatively, a substrate region is formed which is not to be etched and which is to have a desired thickness. In the former case, the etching of the substrate region is monitored, or its thickness is measured (if the etch rate is known), using the inventive etch monitoring technique or the inventive thickness measurement technique to achieve the desired etch depth. In the latter case, the thickness of the substrate region is measured using the inventive thickness measurement technique, and adjusted until the desired thickness is achieved. Once the desired etch depth or the desired thickness has been achieved, the device is completed by a series of conventional steps.

One apparatus for monitoring the etching, or for measuring the thickness, of a substrate region is schematically depicted in Fig. 3. This apparatus includes a source 50 of nonvisible, for example, UV, light, e.g., a mercury arc lamp, as well as a beam splitter 60 which is partially transparent to and partially reflective of, e.g., about 50 percent transparent to and about 50 percent reflective of, the light emitted by the source 50. The apparatus also includes a lens 70 as well as a photodetector 80. In operation, the light emitted by the source 50 is partially transmitted by the beam splitter 60 to the lens 70, which focuses the transmitted light onto the substrate region (or, if the region is within a plasma or wet chemical reactor, onto an optical window of the reactor through which the light impinges the region). Reflected light from the substrate region is then transmitted through the lens 70 to the beam splitter 60, where a portion of this light is reflected onto the detector 80.

Example 1

Both the inventive etch monitoring technique (using, in this instance, UV light) and the conventional etch monitoring technique (using visible light) were used to monitor the etch depth of a substrate which underwent reactive ion etching. The substrate included a 7.6 cm (3-inch) silicon wafer whose flat upper surface was covered by successive layers of HPR-204 resist and silicon dioxide. The resist was deposited by conventional spin-deposition techniques and was baked at 210

degrees Centigrade for about 2 hours. The thickness of the resist, as measured by a Nanospec spectral photometer, was about 1.8 μm . The layer of silicon dioxide was deposited using conventional plasma-assisted chemical vapor deposition techniques, and had a thickness (as determined from the etch monitoring data) of about 0.14 μm .

The apparatus used to reactive ion etch the substrate included a stainless steel, cylindrical reactor chamber which was 61 cm (24 inches) high and 49.3 cm (19 inches) in diameter. Centrally arranged within the reactor chamber was a cylindrical electrode which was 35.6 cm (14 inches) high and hexagonal in cross section. Opposed, parallel sides of the hexagonal electrode were spaced apart by about 15 cm (6 inches).

The substrate was mounted on a side of the hexagon-shaped electrode, and reactive ion etched in a CHF_3 atmosphere for about 10 minutes. During the etching procedure the walls of the reactor chamber were grounded, a 13.56 MHz rf signal was applied to the hexagon-shaped electrode, CHF_3 was flowed into the reactor chamber at 35 ml/min, and the pressure within the reactor chamber was maintained at 1.33 Pa (10 millitorr). The dc bias voltage between the walls of the reactor chamber and the hexagon-shaped electrode was 410 volts, and the power density was 0.07 watts/cm².

During the etching procedure, the etch depth was monitored by shining both UV light (light of wavelength equal to 253.7 nm) and visible light (light of wavelength equal to 632.8 nm) at normal incidence onto the substrate through an optical window of the reactor chamber, and detecting and recording the reflected light. The apparatus involved in etch monitoring, which is schematically depicted in Fig. 3, included a source of UV light 50, i.e., a low pressure mercury arc lamp, Model 11SC-2 sold by the Spectronics Corporation of Westbury, New York, which has a strong, narrow emission line at 253.7 nm. The apparatus also included a beam splitter 60 which consisted of a dielectric film formed on a fused silica substrate. The beam splitter 60, which was oriented at 45 degrees to the light rays emanating from the source 50, was designed to reflect 50 percent, and transmit 50 percent, of any 253.7 nm light incident at an angle of 45 degrees. The UV light transmitted by the beam splitter 60 impinged a fused silica lens 70 which focused this light, through a beam splitter 100 (which was designed to have a high transmittance to 253.7 nm light), onto the substrate through the optical window in the reactor chamber. The UV light reflected from the substrate was transmitted through the beam splitter 100 and lens 70 to the beam splitter 60, where a portion of this light was reflected onto a UV photodetector 80, which consisted of a Model R-166 photomultiplier tube sold by the Hamamatsu Corporation of Middlesex, New Jersey. This detector was sensitive only to wavelengths shorter than about 300 nm.

The etch monitoring apparatus also included a

He-Ne laser 110, Model No. 1103P purchased from the Uniphase Corporation of Mountain View, California, which emitted light of wavelength equal to 632.8 nm. This light successively impinged beam splitters 90 and 100, each oriented perpendicularly to beam splitter 60 and at 45 degrees to the light emanating from the laser 110. The beam splitters 90 and 100 served to transmit (through reflections) a portion of the light emitted by the laser 110 to the substrate through the optical window in the reactor chamber. The beam splitters 90 and 100 also transmitted (through reflections) a portion of the laser light reflected by the substrate to a photodetector 120. The beam splitter 90 was a pellicle beam splitter, Model No. 3743 purchased from the Oriel Corporation of Stratford, Connecticut, which was designed to transmit 50 percent, and reflect 50 percent, of 632.8 nm light incident at an angle of 45 degrees. Beam splitter 100, on the other hand, was a dielectric film formed on a fused-silica substrate, designed to substantially transmit all 253.7 nm light, and reflect 90 percent of 632.8 nm light incident at an angle of 45 degrees. The photodetector 120 was a PIN-5DP detector sold by the United Detector Technology Corporation of Culver City, California.

The intensity of the reflected UV light, as detected by the UV detector 80, was recorded as a function of time by a strip chart recorder, and is displayed in Fig. 4. The intensity of the reflected visible light, as detected by the detector 120, is displayed in the same figure. The portions of the two intensity-time curves corresponding to the etching of the silicon dioxide layer and the etching of the resist layer, have been labeled. As is evident, the UV intensity-time curve corresponding to the silicon dioxide etching includes about 1 3/4 intensity oscillations, and the etch end point (as denoted by an arrow in Fig. 4) occurs at an intensity maximum. (The overall drop in the intensity of the reflected UV light is believed to be caused by the buildup during etching of a UV light-absorbing polymer on the optical window of the reactive ion etching chamber). On the other hand, the visible intensity-time curve corresponding to the silicon dioxide etching only involves about 3/5 of an intensity oscillation, and the position of the etch end point (also denoted by an arrow in Fig. 4) is not at an intensity maximum.

Example 2

The apparatus described in Example 1 was also used to etch, and to monitor the etch depth, of a substrate which included a 7.6 cm (3-inch) silicon wafer whose upper surface supported a first, patterned layer of silicon dioxide, a layer of HPR-204 resist, and a second layer of silicon dioxide. The first silicon dioxide layer, which was grown by conventional thermal oxidation techniques, had a thickness of about 0.35 µm, as measured by a Nanospec spectral photometer. The pattern in this first silicon dioxide layer, which consisted of 5 µm lines and spaces, was formed by conventional lithographic and etching techniques. The thick-

nesses of the HPR-204 resist and the second silicon dioxide layer were the same as those in Example 1.

The intensity-time curves produced by UV and visible light incident on the sample are depicted in Fig. 5. As is evident, the UV intensity-time curve is essentially identical to that in Fig. 4. This shows that the UV reflectance technique is essentially insensitive to the presence of underlying structures or surfaces, e.g., the nonplanar surface associated with the patterned silicon dioxide layer.

Example 3

The apparatus described in Example 1 was again used to etch, and to monitor the etching, of a layer of silicon dioxide covered by a patterned layer of HPR-204 resist, the resist and silicon dioxide layers overlying a 7.6 cm (3-inch) silicon wafer. The silicon dioxide layer, which was grown using conventional thermal oxidation techniques, had a thickness of about 1.8 µm, as measured by the Nanospec spectral photometer. The HPR-204 resist, which had been spin-deposited and baked as described above, had a thickness of about 1.6 µm, as also measured by the Nanospec spectral photometer. The pattern in the resist, which had been formed using the tri-level resist process, consisted of an array of circular openings, each having a diameter of about 3/4 µm, with the center-to-center spacing between adjacent openings being about 1 3/4 µm.

The intensity-time curves resulting from the use of both UV incident light and visible incident light are displayed in Fig. 6. The visible intensity-time curve has slightly more than one intensity oscillation, which is believed to be due almost entirely to the (relatively slow) erosion of the HPR-204 resist. On the other hand, the UV intensity-time curve includes many intensity oscillations due almost entirely to etching of the silicon dioxide layer. As before, the etch end point (denoted by an arrow in Fig. 6) of the silicon dioxide layer occurs at an intensity maximum.

Example 4

The thickness of a layer of plasma-deposited silicon dioxide, overlying a layer of HPR-204 resist (supported by a 7.6 cm (3-inch) silicon wafer), was measured by shining light of wavelength ranging from about 190 nm to about 900 nm onto the silicon dioxide layer, and measuring the intensity of the reflected light as a function of wavelength. The light source and the photodetector were included in a dual beam spectrophotometer, Model 575, purchased from the Perkin-Elmer Corporation of Norwalk, Connecticut. In operation, light emitted by the spectrophotometer impinges both the test sample as well as a reference sample (in this case a 7.6 cm (3-inch) silicon wafer covered by a layer of HPR-204 resist), and the spectrophotometer measures the ratio of the intensities of the light reflected from both samples. The resulting normalized intensities, as a function of wavelength, are depicted in Fig. 7.

For the UV portion of the wavelength range used here, i.e., for wavelengths ranging from about 190 to about 400 nm, the structure of the intensity-wavelength curve is relatively simple. That is, the curve contains relatively few intensity maxima and minima. This is due to the fact that at UV wavelengths, the HPR-204 resist is substantially opaque, and the detected maxima and minimum are determined almost entirely by the thickness of the silicon dioxide layer which is substantially transparent at these wavelengths. On the other hand, at wavelengths beyond the UV range, i.e., for wavelengths longer than 400 nm, the intensity-wavelength curve is relatively complicated. It is believed that this is due to the fact that the HPR-204 resist is no longer opaque, and produces intensities which change rapidly with wavelength.

By using an index of refraction for the silicon dioxide of 1.56, and the first intensity minimum and the first and second intensity maxima contained within the UV wavelength range, the thickness of the silicon dioxide layer was calculated (using the technique described in F. Reizman and W. van Gelder, *Solid State Electronics*, supra, p. 625) to be 0.1193 µm. On the basis of the known plasma deposition rate and deposition time, the silicon dioxide layer was known to be about 0.12 µm thick.

Claims

1. A method for fabricating a device by processing steps carried out on a body, the said processing steps including the step of measuring the thickness of a first region, the said measuring being carried out in the presence of a second region which underlies the first region by illuminating a portion of the first region with light and detecting the intensity of at least a portion of the light reflected from the first region, characterised in that the light used to illuminate the said portion of the first region comprises non-visible light to which the first region is sufficiently transparent to transmit at least five percent of said light incident thereon and the second region is sufficiently opaque to transmit less than five percent of said light incident thereon.

2. A method for fabricating a device by processing steps carried out on a body, the said processing steps including a step of progressively removing material of a first region by etching or milling whilst monitoring the thickness of the first region to control the termination of the said removal process, the said monitoring being carried out in the presence of a second region which underlies the first region, by illuminating a portion of the first region with light and detecting the intensity of at least a portion of the light reflected from the first region, characterised in that the light used to illuminate the said portion of the first region comprises non-visible light to which the first region is sufficiently transparent to transmit at least five percent of said light incident thereon and the second

region is sufficiently opaque to transmit less than five percent of said light incident thereon.

3. A method for fabricating a device by processing steps carried out on a body, the said processing steps including a step of progressively removing material of a first region by etching or milling whilst monitoring the thickness of the first region to control the termination of the said removal process, the said monitoring being carried out in the presence of a second region which partially overlies the first region and acts as a mask during the removal process, by illuminating a portion of the first region with light and detecting the intensity of at least a portion of the light reflected from the first region, characterised in that the light used to illuminate the said portion of the first region comprises non-visible light to which the second region is sufficiently opaque to transmit less than five percent of said light incident thereon.

4. A method as claimed in claim 2, wherein the processing steps include removing said first region subsequent to termination of the progressive removal step.

5. A method as claimed in claim 4, wherein the processing steps include removing said second region subsequent to termination of the progressive removal step.

6. A method as claimed in claim 1 wherein the processing steps include, subsequent to the measuring step, a step of progressively removing at least a portion of said first region by etching or milling, the removal time being determined, in part, by the thickness of said first region as measured in the measuring step.

7. A method as claimed in claim 1, wherein the processing steps include, subsequent to the measuring step, the step of altering a thickness of said first region in response to the measured thickness.

8. A method as claimed in any of the preceding claims wherein said nonvisible light is ultraviolet light.

9. A method for fabricating a device by processing steps carried out on a body, the said processing steps including forming a sacrificial coating including a first region and an underlying second region, progressively removing material of the first region by etching or milling whilst monitoring the thickness of the first region to control the termination of the said removal process by illuminating a portion of the first region with light and detecting the intensity of at least a portion of the light reflected from the first region, the first region being sufficiently transparent to said light to transmit at least five percent of said light incident thereon and subsequently removing the remaining sacrificial coating, characterised in that the second region contains light-absorbing material and the said light is chosen to be light having a wavelength or wavelength range different from that used for alignment purposes and which is absorbed by the light-absorbing material so that the second region is sufficiently opaque to transmit less than five

percent of said light incident thereon.

10. A method for fabricating a device by processing steps carried out on a body, the said processing steps including progressively removing material of a first region by etching or milling whilst monitoring the thickness of the first region to control the termination of the said removal process, the said monitoring being carried out in the presence of a second region which partially overlies the first region and acts as a mask during the removal process, by illuminating a portion of the first region with light and detecting the intensity of at least a portion of the light reflected from the first region and subsequently removing the second region characterised in that the second region contains light-absorbing material and the said light is chosen to be light having a wavelength or wavelength range different from that used for alignment purposes and which is absorbed by the light-absorbing material so that the second region is sufficiently opaque to transmit less than five percent of said light incident thereon.

11. A method as claimed in claim 10, wherein said second region contains less than 25 percent atomic proportion of metallic atoms in their zero oxidation state.

Patentansprüche

1. Verfahren zum Herstellen einer Vorrichtung durch an einem Körper ausgeführte Bearbeitungsschritte, einschließlich des Schrittes einer Messung der Dicke einer ersten Zone, wobei die Messung ausgeführt wird in Gegenwart einer unter der ersten Zone liegenden zweiten Zone durch Beleuchten eines Teils der ersten Zone mit Licht und Feststellen der Intensität wenigstens eines Teils des an der ersten Zone reflektierten Lichtes, dadurch gekennzeichnet, daß das zur Beleuchtung des Teils der ersten Zone benutzte Licht unsichtbares Licht umfaßt, gegenüber dem die erste Zone ausreichend transparent ist, um wenigstens fünf Prozent des hierauf einfallenden Lichtes durchzulassen, und die zweite Zone hinreichend opak ist, um weniger als fünf Prozent des hierauf einfallenden Lichtes durchzulassen.

2. Verfahren zum Herstellen einer Vorrichtung durch an einem Körper ausgeführte Bearbeitungsschritte einschließlich des Schrittes einer progressiven Entfernung von Material einer ersten Zone durch Ätzen oder Abtragen, während die Dicke der ersten Zone zur Steuerung der Beendigung des Materialentfernungsprozesses überwacht wird, wobei die Überwachung ausgeführt wird in Gegenwart einer unter der ersten Zone gelegenen zweiten Zone durch Beleuchten eines Teils der ersten Zone mit Licht und Feststellen der Intensität wenigstens eines Teils des an der ersten Zone reflektierten Lichtes, dadurch gekennzeichnet, daß das zur Beleuchtung des Teils der ersten Zone benutzte Licht unsichtbares Licht umfaßt, gegenüber dem die erste Zone ausreichend transparent ist, um wenigstens fünf Prozent des hierauf einfallenden Lichtes durchzu-

lassen, und die zweite Zone hinreichend opak ist, um weniger als fünf Prozent des hierauf einfallenden Lichtes durchzulassen.

3. Verfahren zum Herstellen einer Vorrichtung durch an einem Körper ausgeführte Bearbeitungsschritte einschließlich des Schrittes einer progressiven Entfernung von Material einer ersten Zone durch Ätzen oder Abtragen, während die Dicke der ersten Zone zur Steuerung der Beendigung des Materialentfernungsprozesses überwacht wird, wobei die Überwachung ausgeführt wird in Gegenwart einer teilweise über der ersten Zone gelegenen zweiten Zone, die als Maske während des Materialentfernungsprozesses wirkt, durch Beleuchten eines Teils der ersten Zone mit Licht und Feststellen der Intensität wenigstens eines Teils des an der ersten Zone reflektierten Lichtes, dadurch gekennzeichnet, daß das zur Beleuchtung des Teils der ersten Zone benutzte Licht unsichtbares Licht umfaßt, gegenüber dem die zweite Zone hinreichend opak ist, um weniger als fünf Prozent des hierauf einfallenden Lichtes durchzulassen.

4. Verfahren wie in Anspruch 2 beansprucht, bei dem die Verfahrensschritte eine Entfernung der ersten Zone, auf die Beendigung des progressiven Materialentfernungschnittes folgend, umfassen.

5. Verfahren wie in Anspruch 4, beansprucht, bei dem die Bearbeitungsschritte eine Entfernung der zweiten Zone, auf die Beendigung des progressiven Materialentfernungschnittes folgend, umfassen.

6. Verfahren wie in Anspruch 1 beansprucht, bei dem die Verfahrensschritte, auf den Messungsschritt folgend, den Schritt einer progressiven Entfernung wenigstens eines Teils der ersten Zone durch Ätzen oder Abtragen umfassen, wobei die Materialentfernungszeit teilweise bestimmt ist durch die Dicke der ersten Zone, wie diese in dem Messungsschritt gemessen wird.

7. Verfahren wie in Anspruch 1 beansprucht, bei dem die Bearbeitungsschritte, auf den Messungsschritt folgend, den Schritt einer Dickenänderung der ersten Zone, ansprechend auf die gemessene Dicke, umfassen.

8. Verfahren wie in einem der vorstehenden Ansprüche beansprucht, bei dem das unsichtbare Licht ultraviolettes Licht ist.

9. Verfahren zum Herstellen einer Vorrichtung durch an einem Körper ausgeführte Bearbeitungsschritte einschließlich Ausbilden einer Oferbeschichtung mit einer ersten Zone und einer darunter liegenden zweiten Zone, progressives Entfernen von Material der ersten Zone durch Ätzen oder Abtragen, während die Dicke der ersten Zone zur Steuerung der Beendigung des Materialentfernungsprozesses überwacht wird durch Beleuchten eines Teils der ersten Zone mit Licht und Feststellen der Intensität wenigstens eines Teils des an der ersten Zone reflektierten Lichtes, wobei die erste Zone gegenüber dem Licht hinreichend transparent ist, um wenigstens fünf Prozent des hierauf einfallenden Lichtes durchzulassen, und nachfolgendes Entfernen der

restlichen Opferbeschichtung, dadurch gekennzeichnet, daß die zweite Zone ein lichtabsorbierendes Material enthält und als Licht ein Licht einer Wellenlänge oder eines Wellenlängenbereiches gewählt wird, das sich diesbezüglich von dem für Ausrichtungszwecke benutzten Licht unterscheidet und in dem lichtabsorbierenden Material absorbiert wird, so daß die zweite Zone ausreichend opak ist, um weniger als fünf Prozent des hierauf einfallenden Lichtes zu übertragen.

10. Verfahren zum Herstellen einer Vorrichtung durch an einem Körper ausgeführte Bearbeitungsschritte einschließlich des Schrittes einer progressiven Entfernung von Material einer ersten Zone durch Ätzen oder Abtragen, während die Dicke der ersten Zone zur Steuerung der Beendigung des Materialentfernungsprozesses überwacht wird, wobei die Überwachung durchgeführt wird in Gegenwart einer zweiten Zone, die teilweise über der ersten Zone liegt und als eine Maske während des Materialentfernungsprozesses wirkt, durch Beleuchten eines Teils der ersten Zone mit Licht und Feststellen der Intensität von wenigstens einem Teil des an der ersten Zone reflektierten Lichtes, und nachfolgendes Entfernen der zweiten Zone, dadurch gekennzeichnet, daß die zweite Zone ein lichtabsorbierendes Material enthält und als das Licht ein Licht einer Wellenlänge oder eines Wellenlängenbereiches gewählt wird, das sich diesbezüglich von dem zu Ausrichtungszwecken benutzten Licht unterscheidet und das durch das lichtabsorbierende Material absorbiert wird, so daß die zweite Zone hinreichend opak ist, um weniger als fünf Prozent des hierauf einfallenden Lichtes durchzulassen.

11. Verfahren wie in Anspruch 10 beansprucht, bei dem die zweite Zone einen Anteil von weniger als 25 Atomprozent an metallischen Atomen in deren Null-Oxidationszustand enthält.

Revendications

1. Un procédé de fabrication d'un dispositif par des étapes de traitement qui sont accomplies sur un article, ces étapes de traitement comprenant l'étape qui consiste à mesurer l'épaisseur d'une première région, cette mesure étant accomplie en présence d'une seconde région qui se trouve au-dessous de la première région, en illuminant une partie de la première région avec de la lumière et en détectant l'intensité d'au moins une partie de la lumière qui est réfléchie par la première région, caractérisé en ce que la lumière qui est utilisée pour illuminer la partie précitée de la première région consiste en une lumière non visible à laquelle la première région est suffisamment transparente pour transmettre au moins cinq pour cent de la lumière qui tombe sur elle, et la seconde région est suffisamment opaque pour transmettre moins de cinq pour cent de la lumière qui tombe sur elle.

2. Un procédé de fabrication d'un dispositif par des étapes de traitement qui sont accomplies sur un article, ces étapes de traitement comprenant une étape qui consiste à enlever progressivement

un matériau d'une première région par gravure ou érosion, tout en contrôlant l'épaisseur de la première région pour commander la terminaison du processus d'enlèvement, ce contrôle étant accompli en présence d'une seconde région qui se trouve au-dessous de la première région, en illuminant une partie de la première région avec de la lumière et en détectant l'intensité d'au moins une partie de la lumière qui est réfléchie par la première région, caractérisé en ce que la lumière qui est utilisée pour illuminer la partie précitée de la première région consiste en une lumière non visible à laquelle la première région est suffisamment transparente pour transmettre au moins cinq pour cent de la lumière qu'elle reçoit, et à laquelle la seconde région est suffisamment opaque pour transmettre moins de cinq pour cent de la lumière qu'elle reçoit.

3. Un procédé de fabrication d'un dispositif par des étapes de traitement qui sont accomplies sur un article, ces étapes de traitement comprenant une étape qui consiste à enlever progressivement un matériau d'une première région par gravure ou érosion, tout en contrôlant l'épaisseur de la première région pour commander la terminaison du processus d'enlèvement, ce contrôle étant accompli en présence d'une seconde région qui recouvre partiellement la première région et qui constitue un masque pendant le processus d'enlèvement, en illuminant une partie de la première région avec de la lumière et en détectant l'intensité d'au moins une partie de la lumière qui est réfléchie par la première région, caractérisé en ce que la lumière qui est utilisée pour illuminer la partie précitée de la première région consiste en une lumière non visible à laquelle la seconde région est suffisamment opaque pour transmettre moins de cinq pour cent de la lumière qu'elle reçoit.

4. Un procédé selon la revendication 2, dans lequel les étapes de traitement comprennent l'enlèvement de la première région à la suite de la terminaison de l'étape d'enlèvement progressif.

5. Un procédé selon la revendication 3, dans lequel les étapes de traitement comprennent l'enlèvement de la seconde région à la suite de la terminaison de l'étape d'enlèvement progressif.

6. Un procédé selon la revendication 1, dans lequel les étapes de traitement comprennent, à la suite de l'étape de mesure, une étape d'enlèvement progressif d'au moins une partie de la première région par gravure ou érosion, la durée d'enlèvement étant déterminée en partie par l'épaisseur de la première région, telle qu'elle mesurée dans l'étape de mesure.

7. Un procédé selon la revendication 1, dans lequel les étapes de traitement comprennent, à la suite de l'étape de mesure, l'étape qui consiste à modifier l'épaisseur de la première région sous la dépendance de l'épaisseur mesurée.

8. Un procédé selon l'une quelconque des revendications précédentes, dans lequel la lumière non visible est de la lumière ultraviolette.

9. Un procédé de fabrication d'un dispositif par des étapes de traitement qui sont accomplies sur

un article, ces étapes de traitement comprenant la formation d'un revêtement perdu qui comprend une première région et une seconde région sous-jacente, l'enlèvement progressif du matériau de la première région par gravure ou érosion, tout en contrôlant l'épaisseur de la première région pour commander la terminaison du processus d'enlèvement, en illuminant une partie de la première région avec de la lumière, et en détectant l'intensité d'au moins une partie de la lumière qui est réfléchie par la première région, la première région étant suffisamment transparente à la lumière précitée pour transmettre au moins cinq pour cent de la lumière qui tombe sur elle, et ensuite l'enlèvement du revêtement perdu restant, caractérisé en ce que la seconde région contient une matière absorbant la lumière et la lumière précitée est choisie de façon à être de la lumière ayant une longueur d'onde ou une plage de longueurs d'onde différente de celle qui est utilisée dans un but d'alignement, et qui est absorbée par la matière absorbant la lumière, de façon que la seconde région soit suffisamment opaque pour transmettre moins de cinq pour cent de la lumière qui tombe sur elle.

10. Un procédé de fabrication d'un dispositif par des étapes de traitement qui sont accomplies

sur un article, ces étapes de traitement comprenant l'enlèvement progressif du matériau d'une première région, par gravure ou érosion, tout en contrôlant l'épaisseur de la première région pour commander la terminaison du processus d'enlèvement, ce contrôle étant accompli en présence d'une seconde région qui recouvre partiellement la première région et qui constitue un masque pendant le processus d'enlèvement, en illuminant une partie de la première région avec de la lumière et en détectant l'intensité d'au moins une partie de la lumière qui est réfléchie par la première région, et ensuite l'enlèvement de la seconde région caractérisé en ce que la seconde région contient une matière absorbant la lumière, et la lumière précitée est choisie de façon à être de la lumière ayant une longueur d'onde ou une gamme de longueurs d'onde différente de celle qui est utilisée dans un but d'alignement, et qui est absorbée par la matière absorbant la lumière, de façon que la seconde région soit suffisamment opaque pour transmettre moins de cinq pour cent de la lumière qui tombe sur elle.

11. Un procédé selon la revendication 10, dans lequel la seconde région contient des atomes métalliques dans leur état d'oxydation zéro, dans une proportion atomique inférieure à 25%.

30

35

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60

65

12

FIG. 1

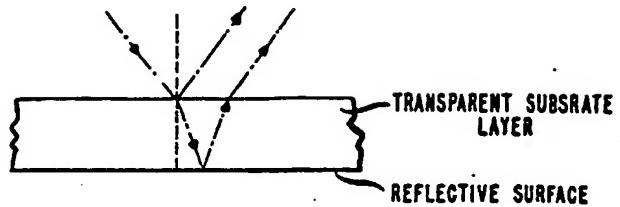


FIG. 2

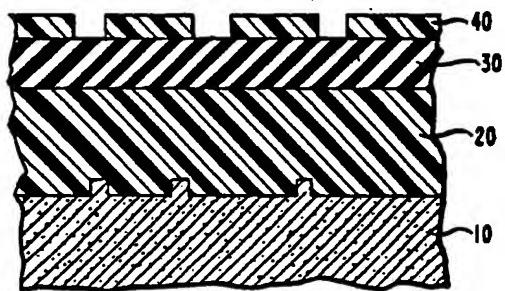


FIG. 6

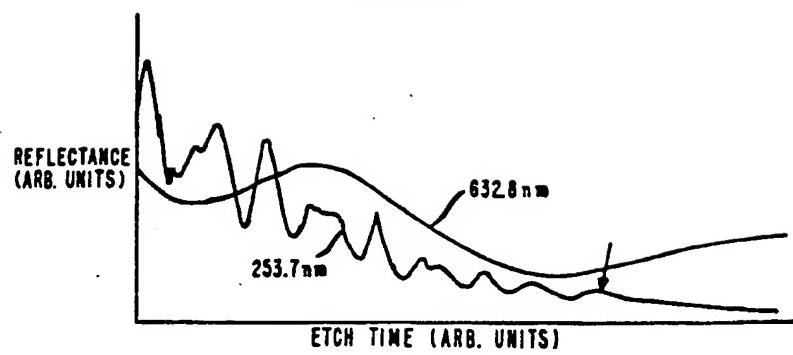


FIG. 3

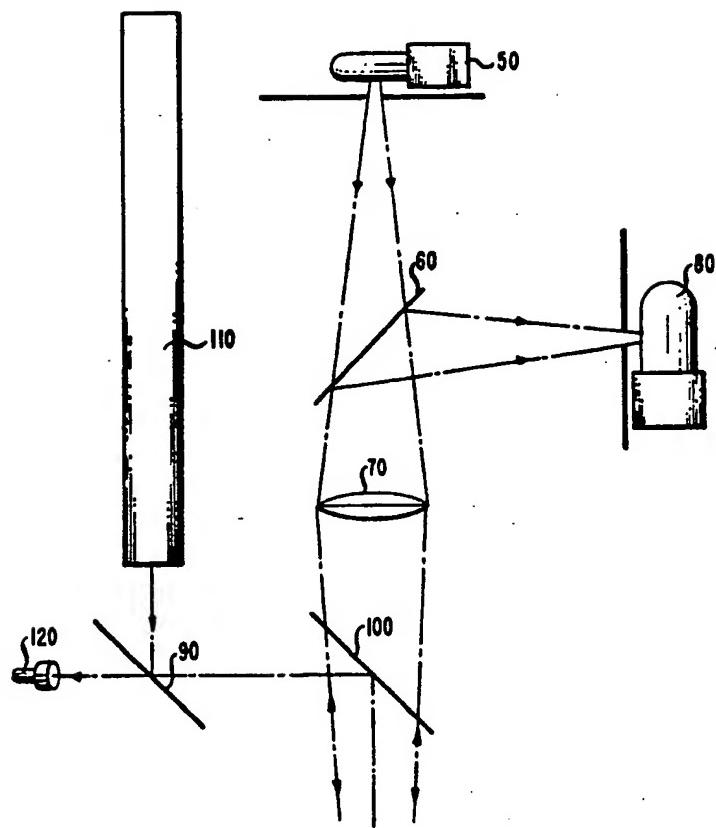


FIG. 5

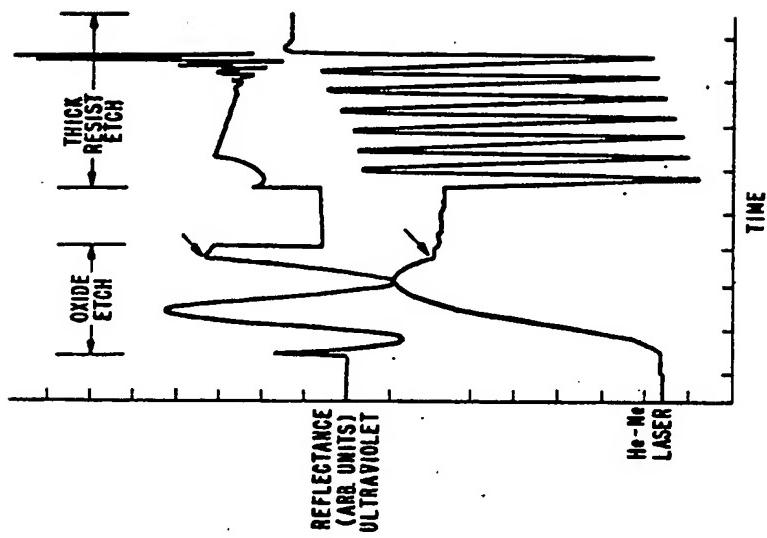
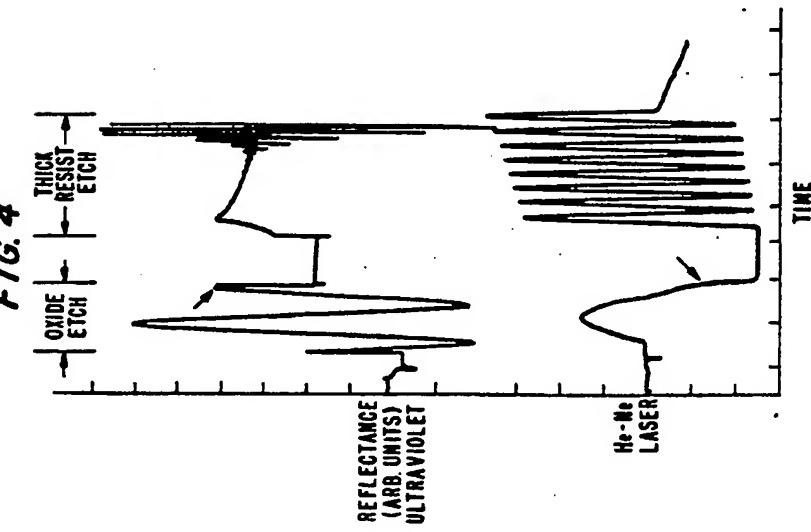


FIG. 4



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FIG. 7

